

HOT SALT STRESS CORROSION CRACKING OF  
SHEET MATERIALS FOR A SUPERSONIC TRANSPORT

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# HOT SALT STRESS CORROSION CRACKING OF SHEET MATERIALS FOR A SUPERSONIC TRANSPORT

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(Presented at Symposium on Corrosion at AIME Annual Meeting, February 19-23, 1967, Los Angeles.)

An extensive investigation of hot salt stress corrosion of sheet materials for a supersonic transport began at the NASA Langley Research Center in 1962. Studies included the relative susceptibility of titanium alloy and stainless steel sheet materials, environmental effects, crack initiation, residual strength, the corrosion mechanism, and protective treatments. Many of the results have been reported previously. This talk will briefly review some of the findings and discuss current results.

The sheet materials are listed in the first figure. The stainless steels (on right) were found to be resistant to hot salt stress corrosion up to 10,000 hours at 550° F when stressed at 100 ksi. On the other hand, the titanium alloys were susceptible to this type of corrosion - some after relatively short exposures under these conditions. The alloys are shown in the order of their resistance with Ti-8Al-1Mo-1V the least and Ti-4Al-3Mo-1V the most resistant. Note that alloys with low aluminum content are less susceptible. Ti-679 (with 2 Al) not shown is also very resistant. Most of our corrosion research was carried out on Ti-8Al-1Mo-1V in the duplex anneal condition which was favored for the supersonic transport until recently when its susceptibility to aqueous stress corrosion was discovered.

In order to carry out this research, new specimens and testing techniques were devised which eliminated the need for fixtures and provided a rapid method for studying stress corrosion damage. Large numbers of small specimens were salt coated and exposed up to 15,000 hours at 400° to 600° F and then tested at room temperature to determine the amount of stress corrosion cracking.

The two types of specimens are illustrated in figure 2. The self-stressed specimen (above) consists of two 1/4-inch strips 4-inches long which are initially bent up at the ends and then spotwelded together to induce uniform bending stresses in the curved portion. The residual stress specimens (below) are brake formed from small pieces of sheet (about 1/2 in. by 2 in.) so as to produce radii of 1/8 inch (on left) and from 1/4 to 2-1/2 inches (on right). Residual tensile stresses are developed on the inside of the bend. After the exposure, the stress corrosion damage is determined from the decrease in the shortening  $\delta$  in the compression test (above) and from the decrease in the bend ductility in the bend test (below). Details of the testing and analysis will not be discussed here.



To shed some light on the mechanism, studies were made of various environmental factors (besides stress, temperature, and time) which might affect the process. These factors are noted on figure 3. The type and condition of the salt are important. NaCl is more corrosive than sea salt,  $\text{CaCl}_2$  or  $\text{MgCl}_2$  (the least corrosive) and thin coatings are worse than thick, except perhaps at very high temperatures. Oxygen appears essential for corrosion, and a reduction in pressure (and oxygen) such as at high altitudes reduces corrosion. The effect of moisture is still unresolved although there is evidence that it is necessary and accelerates the process. The effect of velocity under dynamic environmental conditions such as occur in supersonic flight is still unknown. A current NASA contract, in which stress corrosion specimens will be subjected to 600° F under simulated Mach 2.5 and 70,000-foot altitude conditions, may throw some light on this question, and show whether static laboratory tests are applicable. Lastly, the effect of thermal cycles in reducing stress corrosion is important and appears to be beneficial, but relatively little information is available.

One of the characteristics of NaCl which may be a factor in the corrosion process is the ability of salt to retain inclusions of  $\text{H}_2\text{O}$  even under supposedly dry conditions. The loss of entrapped moisture in reagent grade NaCl when heated under low pressure conditions is shown in figure 4. In the as-received condition (upper curve) moisture is not driven off until above about 450° C, and in the recrystallized condition (lower curve) until above about 200° C. Consequently, even under supposedly dry elevated-temperature conditions such as found at 70,000 feet, some moisture may be present in the salt which may be instrumental in accelerating corrosion. However, a general understanding of the role of moisture and the corrosion mechanism is still lacking in spite of intensive research by many laboratories.

For structural applications in which the possibility of stress corrosion cracking must be considered, the determination of stress-temperature-time thresholds for crack initiation is essential. Some long-time data obtained from numerous tests of the small self-stressed specimens are now available and summarized in figure 5. These preliminary curves indicate the approximate threshold stress for crack initiation in Ti-8Al-1Mo-1V for exposures from 100 to 10,000 hours in the 400° to 600° F range. To the left of any particular curve, no stress corrosion occurs; to the right stress corrosion cracking can be expected. No stress corrosion cracking was obtained at 400° F. At 550° F, however, the stress for crack initiation in 10,000 hours is about 25 ksi for this susceptible alloy. Ti-6Al-4V is much more resistant. Tests at 550° F have shown no stress corrosion cracking even after 3200 hours at 50 ksi. As Ti-6Al-4V will now be used for a Mach 2.7 rather than a Mach 3 supersonic transport, hot stress corrosion is no longer considered a potential structural problem for this application on the basis of such laboratory tests. Operational conditions of about 25 ksi and 450° F are well within the no corrosion area, even for Ti-8Al-1Mo-1V. Realistic environmental tests are needed, however, to validate such test results.

Stress corrosion data are amenable to parameter analysis as illustrated in figure 6. This shows master curves for crack initiation (solid curve)

and for corrosion strength (dashed curve), employing the Larson-Miller time-temperature parameter (the time  $t$  is in hours and the temperature  $T$  is in  $^{\circ}\text{R}$ ). Again, the region to the left of each curve indicates no stress corrosion cracking (solid curve) or no stress corrosion failure (dashed curve). The correlation of the test points at 50 ksi with the solid curve for crack initiation is acceptable at 500 $^{\circ}$  F and above. The correlation at 25 ksi is limited to 550 $^{\circ}$  and 600 $^{\circ}$  F, however. The relation between the solid curve for crack initiation and the dashed curve for stress corrosion rupture or failure appears reasonable. The time difference between crack initiation and failure at 40 ksi is about 90 hours. This type of analysis implies that some kind of rate process holds for crack initiation as well as for corrosion strength.

In a related program, tensile creep specimens of Ti-8Al-1Mo-1V were exposed at 500 $^{\circ}$  to 600 $^{\circ}$  F while under 50 ksi stress in order to study both the magnitude of the stress corrosion cracking and its effect on the tensile properties. Figure 7 illustrates the characteristic small size and large number of surface cracks obtained in Ti-8Al-1Mo-1V in the longitudinal and transverse directions after an 800-hour exposure at 550 $^{\circ}$  and 600 $^{\circ}$  F with a 50 ksi stress. The load is applied horizontally in these photographs and the cracks are normal to the applied load. The magnification is about 5. The cracks are usually more visible near the edge of the fracture (on left side of each micrograph). Note the very large number of fine cracks in the upper right. Such characteristic stress corrosion cracks are generally not readily seen without some magnification.

Quantitative data on the crack penetration measurements for Ti-8Al-1Mo-1V are shown in figure 8. The bar graphs give the maximum crack depths in the longitudinal and transverse directions at 550 $^{\circ}$  and 600 $^{\circ}$  F after exposures up to 800 hours with a 50 ksi stress. About a dozen measurements were obtained for each test condition. Crack depths up to 10 to 15 mils were obtained at 550 $^{\circ}$  and up to 15 to 20 mils at 600 $^{\circ}$  F. Crack depths were greatest in the transverse direction. As the sheet was only 0.05-inch thick, the cracks penetrated nearly half way through the sheet in some cases.

If failure or fracture does not occur because of stress corrosion cracking during exposure, the question arises as to their effect on the tensile properties of the material. Figure 9 shows some data on residual strength and elongation for Ti-8Al-1Mo-1V after exposure up to 1000 hours at 500 $^{\circ}$  and 600 $^{\circ}$  F. A large reduction in strength above 500 $^{\circ}$  F was obtained for Ti-8Al-1Mo-1V and several other alloys (Boeing data) after being stressed at 40 ksi for 1000 hours. A much smaller reduction in strength was found for Ti-8Al-1Mo-1V (NASA data) after an 800-hour exposure for a 50 ksi stress. (L and T refer to the longitudinal and transverse directions.) On the other hand, the reduction in elongation after exposure at 500 $^{\circ}$  F and above after 800 hours (NASA data) is very large, indicating severe embrittlement. Consequently, stress corrosion cracking can have very detrimental effects on the tensile properties.

In view of the susceptibility of titanium alloys to hot salt stress corrosion, an investigation was made of the effectiveness of various protective treatments. The approaches taken are outlined in figure 10. These consisted of platings, coatings, and chemical and mechanical surface treatments. The specimens of Ti-8Al-1Mo-1V were stressed up to 65 ksi and exposed up to 15,000 hours at 600° F. A nickel plating was effective, but after 10,000 hours diffusion appeared to reduce the substrate ductility. The electro-phoretically deposited aluminum coating proved satisfactory for at least 10,000 hours. The polyimide coating prevented salt stress corrosion initially, but after about 750 hours, it tended to peel off. Anodizing treatments were ineffective, but mechanical treatments such as vibratory and glass-bead peening were effective. A final report on protective treatments is in preparation.

Protection against stress corrosion is achieved in mechanical treatments by inducing residual compressive stresses in the surface of the specimen. Figure 11 shows the magnitude of the residual surface stresses in Ti-8Al-1Mo-1V and Ti-6Al-4V obtained by glass-bead peening and a vibratory cleaning treatment for various times. The residual compressive stress induced by glass-bead peening (left) is about 110 ksi for both materials. The size of the beads and the peening time are not critical. The vibratory treatment (right) produced much lower surface stresses and likewise was not time critical. Such residual compressive stresses oppose the applied tensile stresses and thus reduce the possibility of stress corrosion cracking.

Residual surface stresses, however, may not afford long-time protection against stress corrosion if relaxation occurs during elevated-temperature exposure. The deterioration of residual surface stresses in Ti-6Al-4V, induced by mechanical surface treatments, is shown in figure 12. The residual stress remaining (in percent of the original induced surface stress) is given for glass-bead peening (left) and for the vibratory treatment (right) for exposures up to 1000 hours and temperatures up to 800° F. (The initial stresses are shown parenthetically.) The deterioration in the surface stress begins as low as 200° F for glass-bead peening and 400° F for the vibratory treatment. Exposure effects are pronounced even for exposures as low as 10 hours. Consequently, mechanical treatments cannot be relied upon for protection for long-time applications above about 400° F if tensile stresses are large.

In conclusion I would like to make some comments relative to the research status of the hot salt stress corrosion problem for titanium alloys. At the present time extensive laboratory data are available on commercial titanium alloys and the problem is well categorized. Some environmental effects are known, but the applicability of laboratory data to realistic dynamic environmental conditions has not been established. Continued research on the corrosion mechanism is required because the process is

still essentially undefined. Although this type of corrosion does not pose a problem for the sheet material in a Mach 2.7 supersonic transport, it will be of concern for future high-speed aircraft or high performance engines utilizing titanium alloys. Finally, various surface treatments can be employed if required which are effective in preventing or reducing stress corrosion.

MATERIALS

<u>TITANIUM ALLOYS</u>	<u>STAINLESS STEELS</u>
Ti-8Al-1Mo-1V	AM 350 CRT
Ti-6Al-4V	PH 15-7Mo
Ti-13V-11Cr-3Al	PH 14-8Mo
Ti-4Al-3Mo-1V	AISI 301

Figure 1

# STRESS CORROSION SPECIMEN AND TEST PROCEDURE

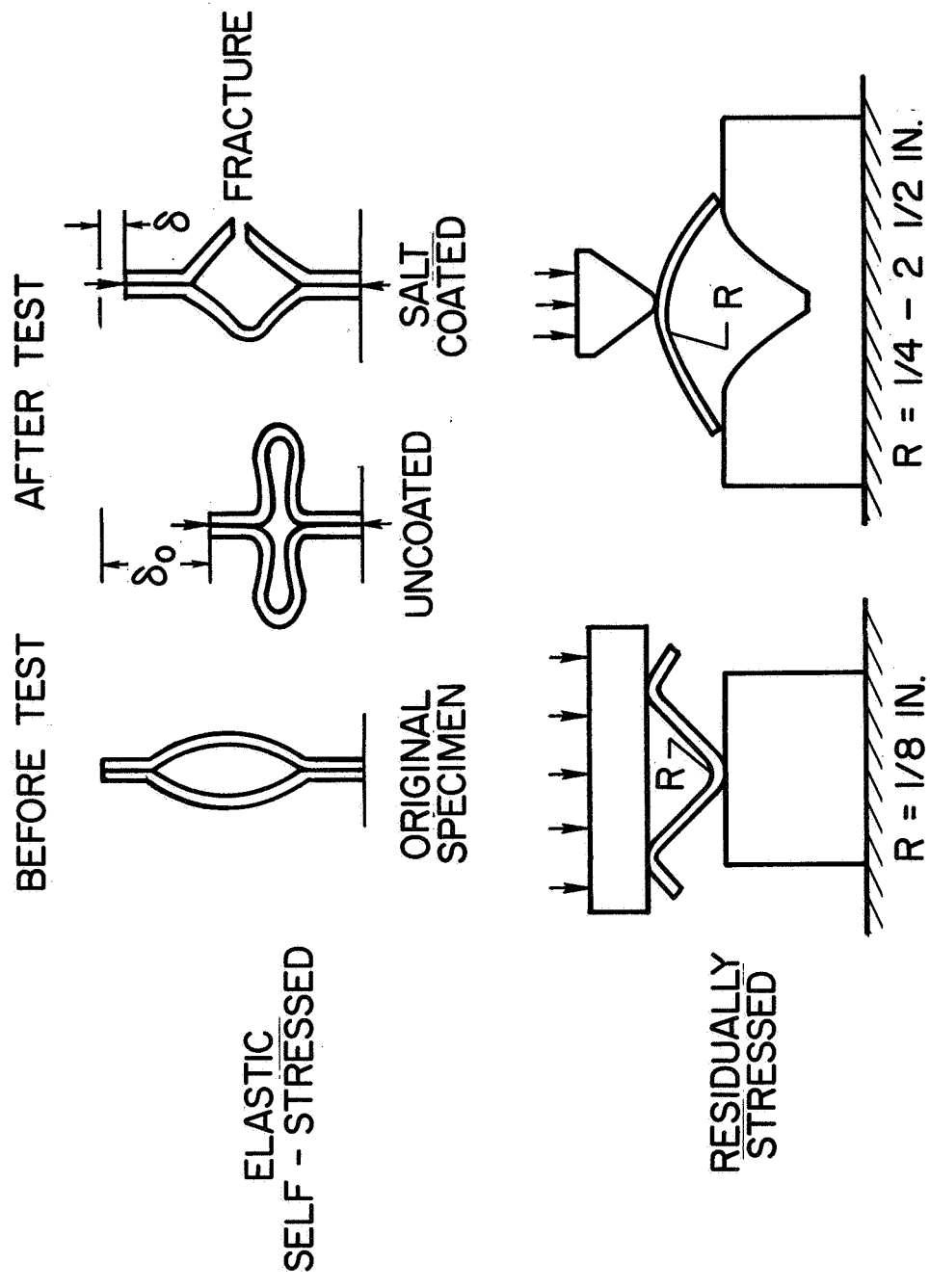


Figure 2



## ENVIRONMENTAL FACTORS

<u>ITEM</u>	<u>REMARKS</u>
SALT	IMPORTANT
OXYGEN, PRESSURE	IMPORTANT
MOISTURE	UNKNOWN
VELOCITY	UNKNOWN
THERMAL CYCLES	IMPORTANT

Figure 3

# LOSS OF ENTRAPPED MOISTURE IN REAGENT GRADE NaCl

PRESSURE,  $1 \times 10^{-5}$  TORR,  
HEATING RATE,  $4^{\circ}$  C/MIN

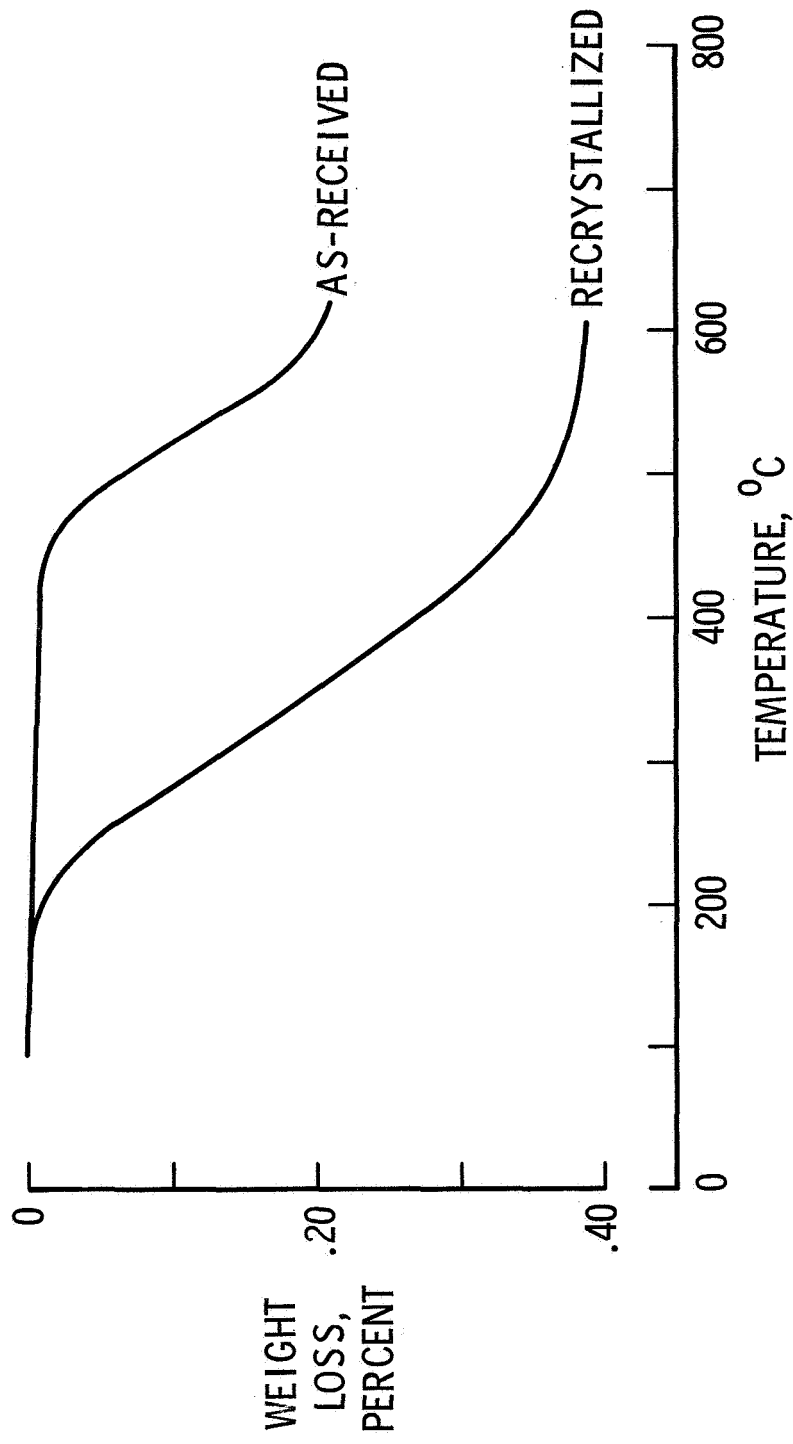


Figure 4

# THRESHOLD STRESS FOR CRACK INITIATION

Ti-8Al-1Mo-1V

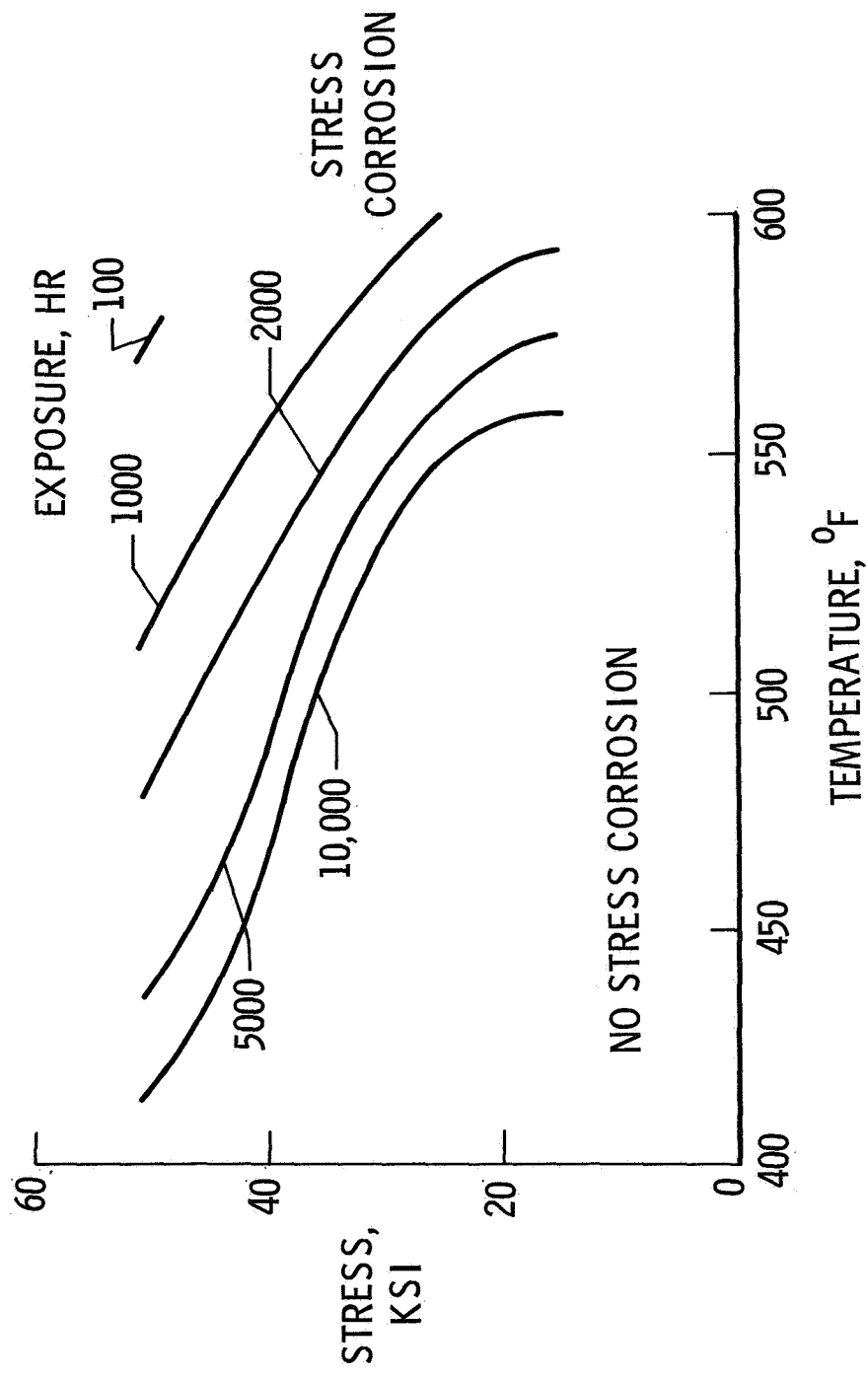


Figure 5

# MASTER CURVE FOR CRACK INITIATION

Ti-8Al-1Mo-IV

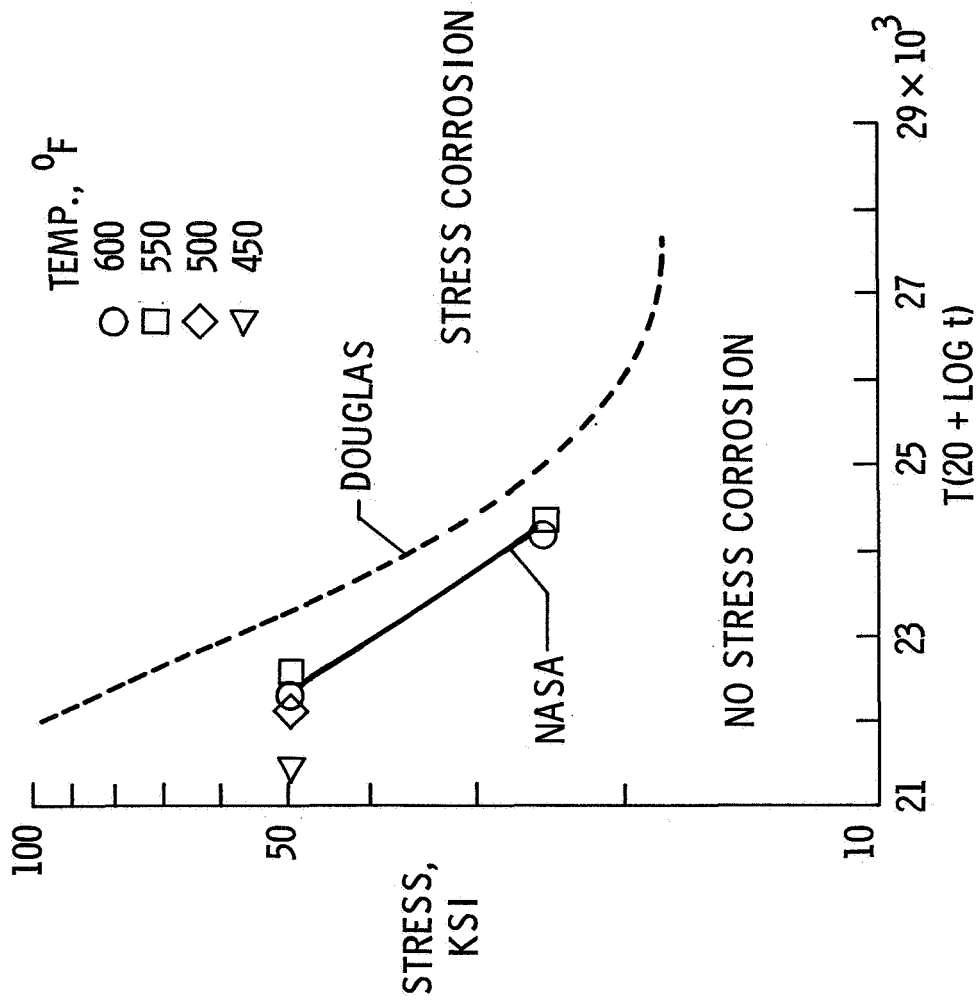
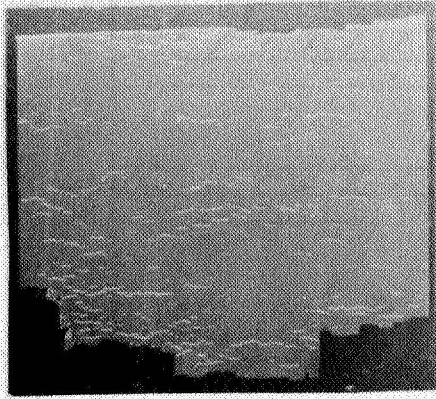
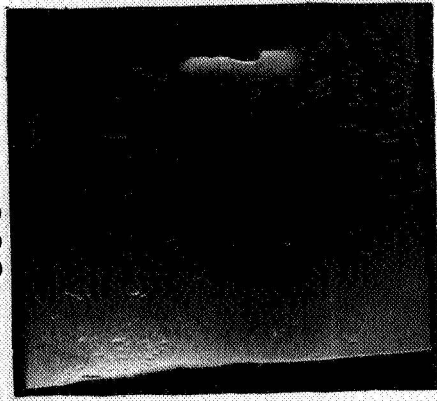


Figure 6

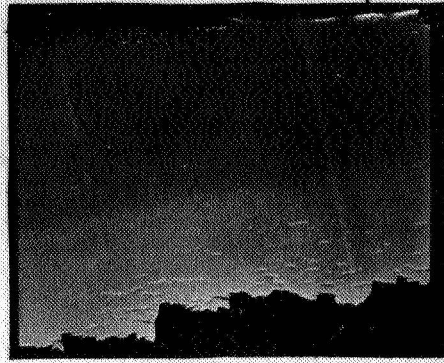
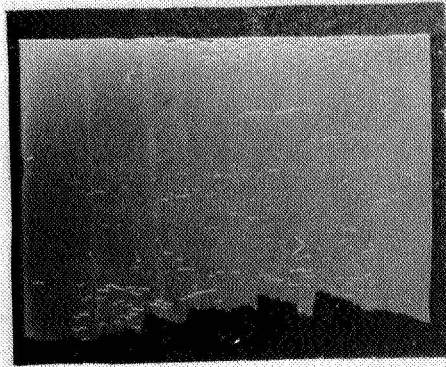
**STRESS CORROSION CRACKS IN Ti-8Al-1Mo-1V**  
**800 HOUR EXPOSURE, 50 KSI STRESS**

**550° F**

**600° F**



**LONGITUDINAL**



**TRANSVERSE**

**Figure 7**

# CRACK PENETRATION FOR Ti-8Al-1Mo-IV EXPOSED UP TO 800 HOURS, 50 KSI STRESS

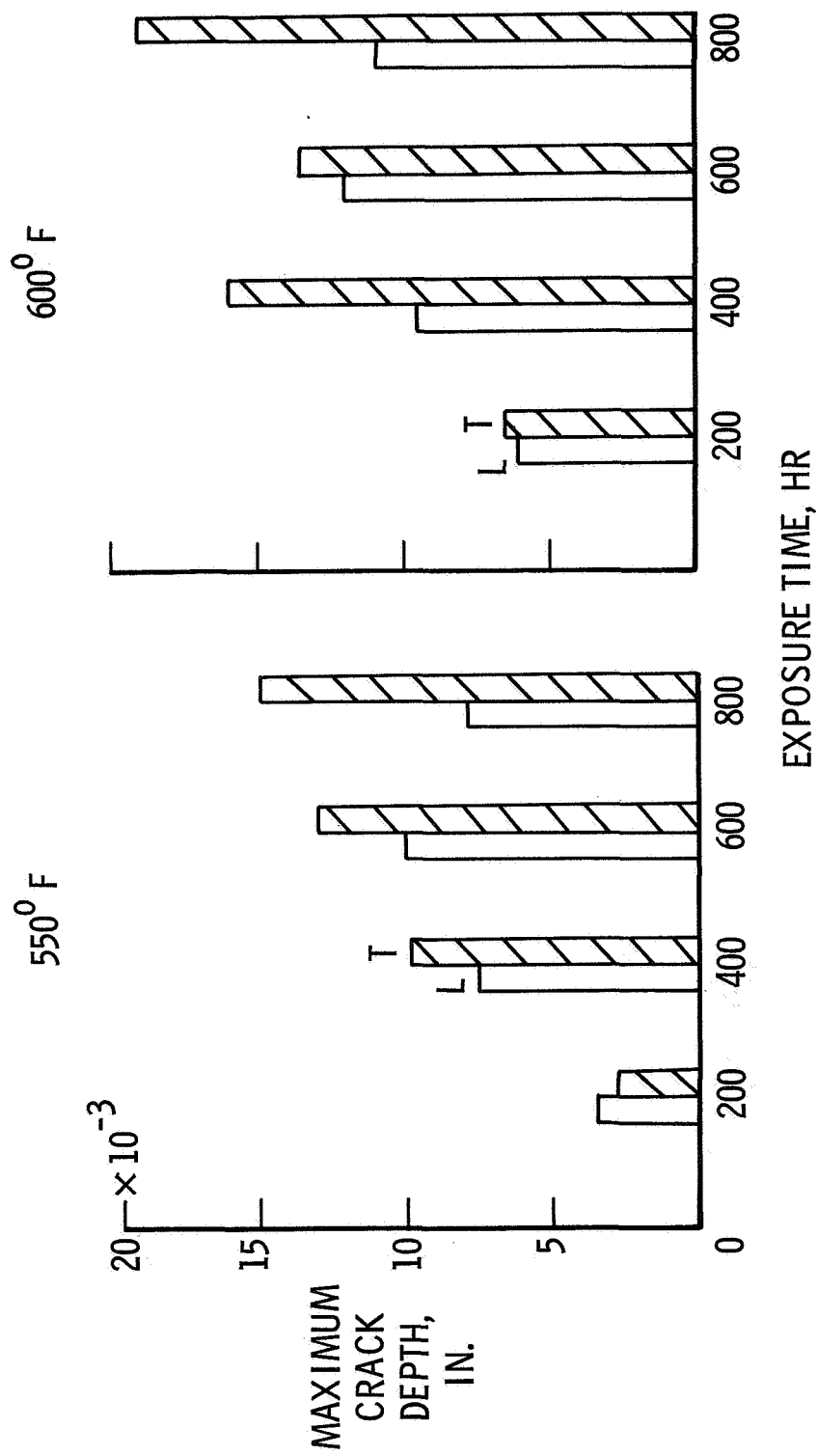


Figure 8

# RESIDUAL STRENGTH AND ELONGATION

Ti-8Al-1Mo-1V

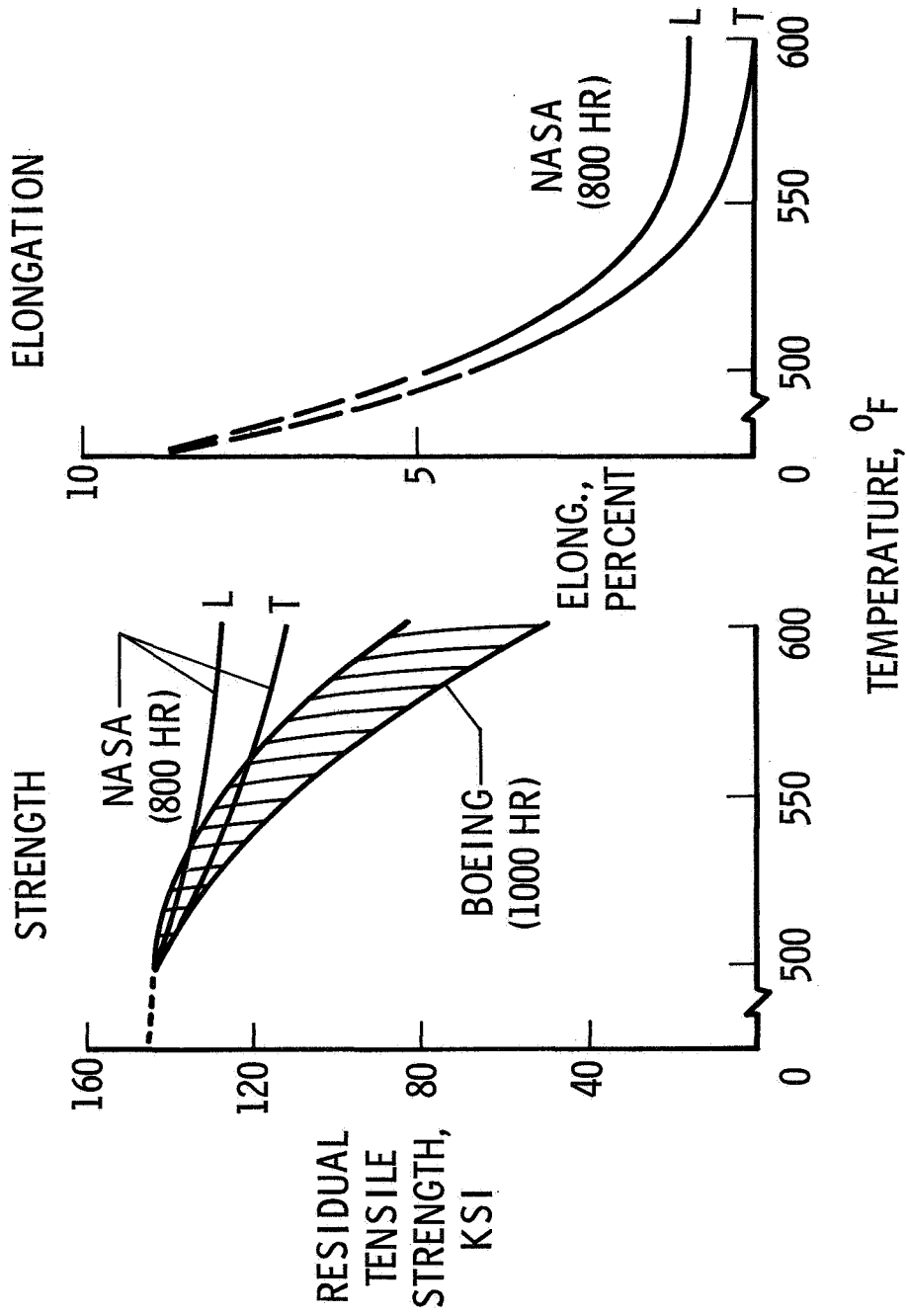


Figure 9

# PROTECTIVE TREATMENTS

PLATINGS

COATINGS

SURFACE TREATMENTS

CHEMICAL

MECHANICAL

Figure 10



# RESIDUAL SURFACE STRESSES

Ti-8Al-1Mo-1V and Ti-6Al-4V

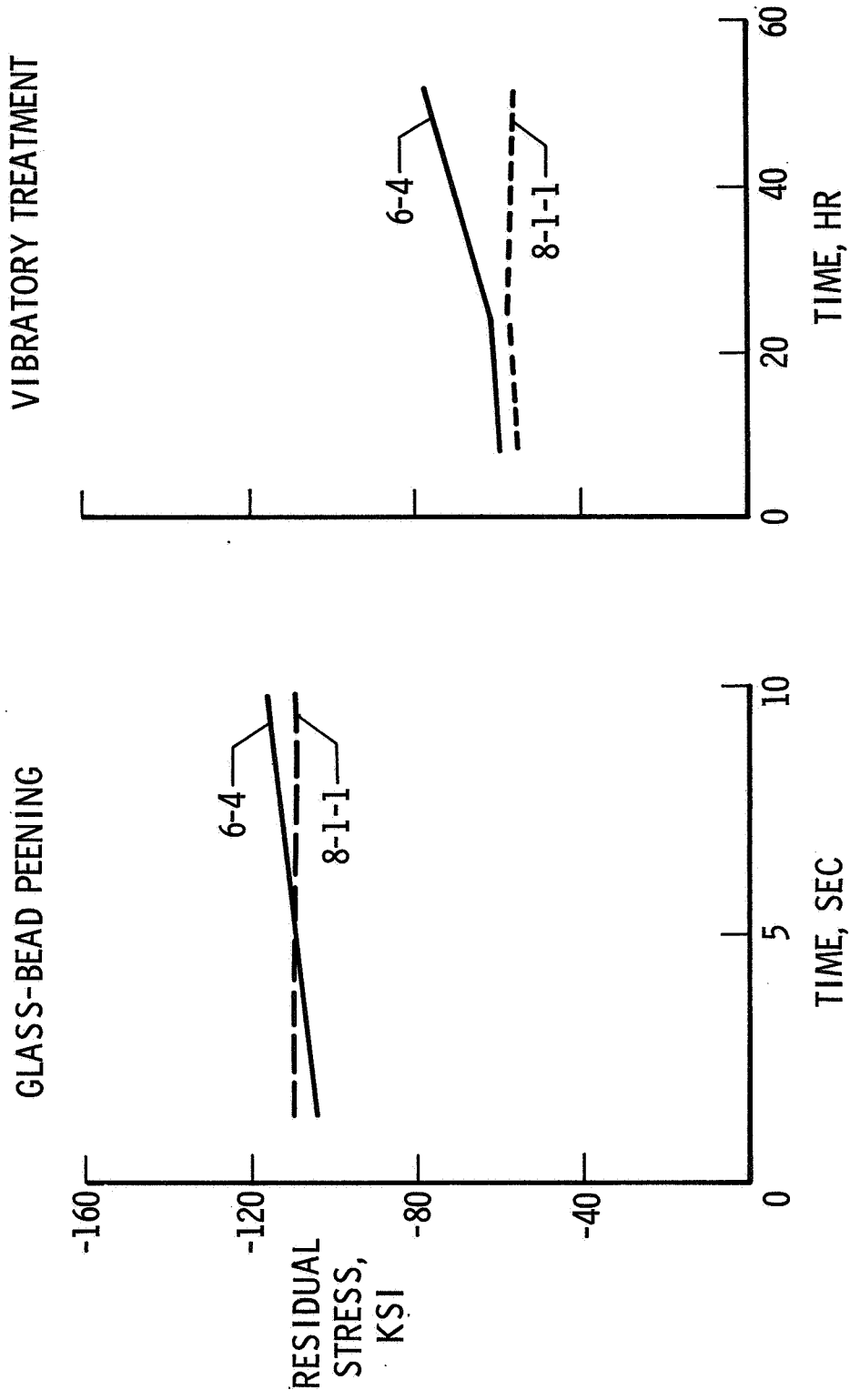


Figure 11

# RESIDUAL STRESS AFTER EXPOSURE

Ti-6Al-4V

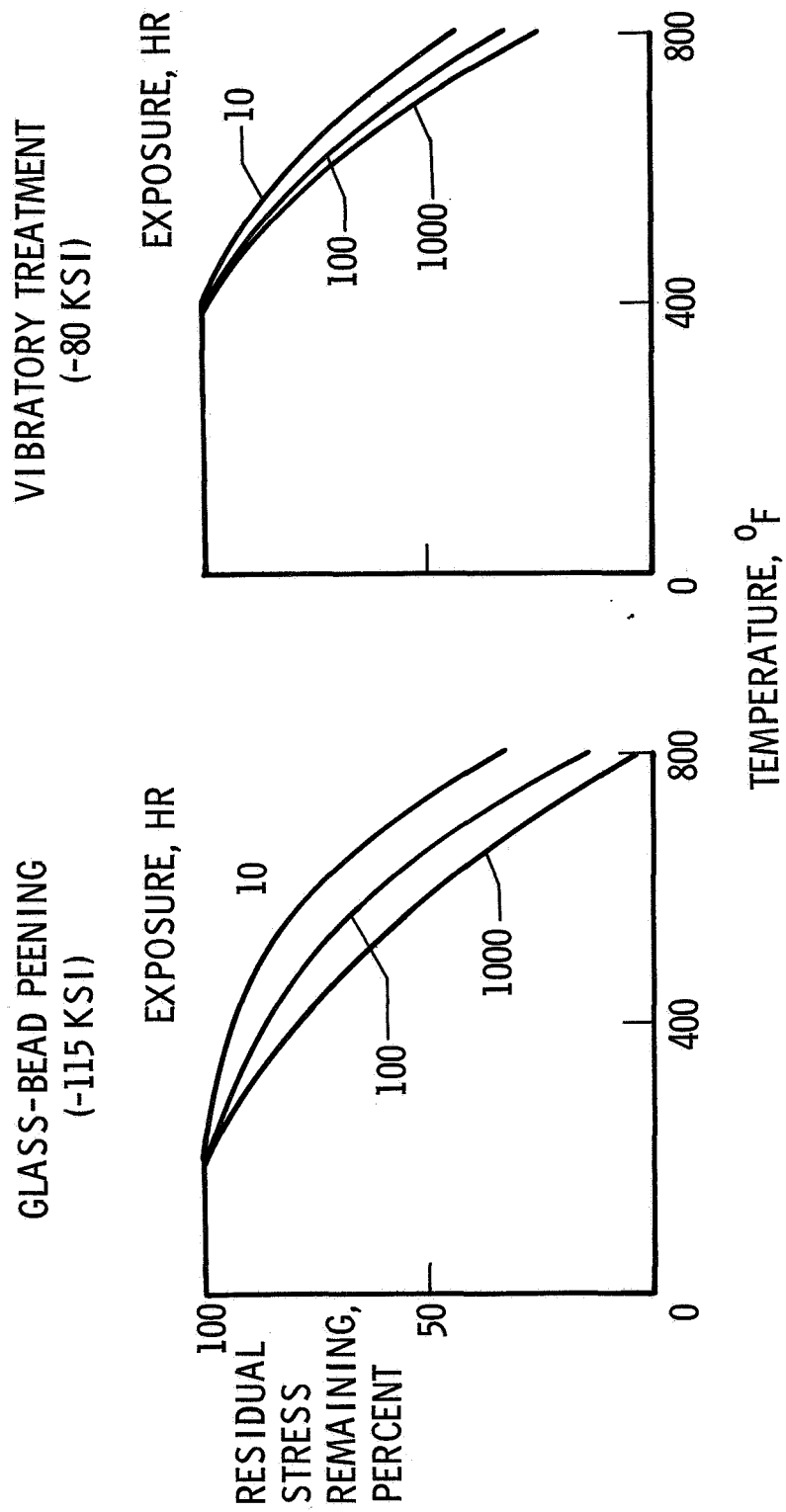


Figure 12